Lacquer formulations

The invention relates to lacquer formulations.

Lacquer formulations are used for sealing or coating surfaces. In the case of special surfaces such as, for example, surfaces of parquet floors or other floors, or motor vehicle bodies, special scratch resistance is desired.

DE 198 11 790 A1 describes improving the scratch resistance of transparent lacquer binders by the use of pyrogenic hydrophilic and/or hydrophobic silicas. In that case, the silica is incorporated into the binder using a special process.

It is a disadvantage that, with the known silicas, despite the use of a special dispersing device, the rheology of the lacquer system is affected to a very great extent and highly flawed lacquer surfaces are obtained (orange-peel effect).

The invention provides lacquer formulations containing

- from 5 to 99.5 wt.%, preferably from 20 to 80 wt.%, particularly preferably from 35 to 70 wt.%, of solids of a polymer component or of a mixture of two or more physically or chemically crosslinking polymer components (binder components and hardener components),
- from 0 to 80 wt.%, preferably from 20 to 70 wt.%, particularly preferably from 30 to 55 wt.%, of a low molecular weight component acting as solvent or of a mixture of such low molecular weight components,
- from 0.5 to 50 wt.%, preferably from 1 to 25 wt.%, particularly preferably from 2 to 10 wt.%, of a silanised, structurally modified pyrogenic silica to the

surface of which there are attached alkylsilyl groups (SiC_nH_{2n+1}, where n = from 2 to 18), preferably octylsilyl and/or hexadecylsilyl groups, or dimethylsilyl and/or monomethylsilyl groups, preferably dimethylsilyl groups,

- from 0 to 10 wt.% of commercially available additives which have a positive effect on the properties of a lacquer, such as, for example, the rheological behaviour of the liquid system, stabilisation and dispersibility of fillers and pigments, appearance and flow of the lacquer surface.

As binders there may be used the resins conventional in lacquers and coatings technology, as are described, for example, in Lackharze, Chemie, Eigenschaften und Anwendungen, Eds. D. Stoye, W. Freitag, Hanser Verlag, Munich, Vienna 1996. Examples which may be mentioned include, *inter alia*, polymers and copolymers of (meth)acrylic acid and their esters, optionally carrying further functional groups, with further olefinically unsaturated compounds, such as, for example, styrene; also polyether, polyester, polycarbonate, polyurethane and epoxy resins, as well as any desired mixtures of those polymers.

As polymeric organic compounds there are preferably used polymers carrying hydroxyl groups, for example polyacrylate, polyester, polycaprolactone, polyether, polycarbonate and polyurethane polyols and hydroxy-functional epoxy resins, as well as any desired mixtures of those polymers. As particularly preferred polymeric organic compounds there are used aqueous or solvent-containing or solvent-free polyacrylate and polyester polyols, as well as any desired mixtures thereof. Suitable polyacrylate polyols are copolymers of hydroxyl-group-containing monomers with other olefinically unsaturated monomers, such as, for example, esters of (meth)acrylic acid, styrene, α-methylstyrene, vinyltoluene, vinyl esters, maleic and

fumaric acid mono- and di-alkyl esters, α-olefins and other unsaturated oligomers and polymers.

Particularly suitable polyacrylate polyols have an average weight-average molecular weight, which can be determined by means of gel permeation chromatography (standard polystyrene), of from 2000 to 10,000 g/mol., preferably from 2500 to 50,000 g/mol. and particularly preferably from 3100 to 40,000 g/mol., a glass transition temperature TG of from -50°C to +100°C, preferably from -40°C to +90°C and particularly preferably -30°C (to +80°C), an acid number of < 30 mg KOH/g, preferably < 25 mg KOH/g, and a hydroxyl group content of from 0.5 to 14.0 wt.%, preferably from 0.5 to 10.0 wt.% and particularly preferably from 1.0 to 8.0 wt.%, and consist of

a) from 0 to 70 parts by weight, preferably from 5 to 70 parts by weight, of at least one unsaturated, aromatic monomer, such as styrene, methylstyrene or vinyltoluene,

b) from 0 to 70 parts by weight, preferably from 5 to 70 parts by weight, of at least one (cyclo)aliphatic ester of acrylic and/or methacrylic acid having from 1 to 18 carbon atoms in the (cyclo)alkyl radical,

c) from 4 to 95 parts by weight, preferably from 10 to 60 parts by weight, of at least one hydroxyalkyl ester of acrylic and/or methacrylic acid having from 2 to 4 carbon atoms in the hydroxyalkyl radical, and/or addition products of monoepoxides with acrylic and/or methacrylic acid,

d) from 0 to 10 parts by weight, preferably from 0.1 to 5 parts by weight, of at least one monoolefinically unsaturated mono- or di-carboxylic acid having from 3 to 7 carbon atoms, and/or at least one maleic acid or

fumaric acid semi-ester having from 1 to 14 carbon atoms in the alcohol radical, and

e) from 0 to 30 parts by weight, preferably from 0 to 20 parts by weight, of further copolymerisable, olefinically (also polyolefinically) unsaturated monomeric and/or polymeric compounds.

The described hydroxyl-functional polyols can be used in both one-component and two-component lacquer systems together with the hardeners conventional in lacquers and coatings technology. There come into consideration as hardeners therefor, for example, polyisocyanates or polyisocyanates blocked with cleaving agents such as methyl ethyl ketoxime, caprolactam, malonic acid esters, triazole or 2,5-dimethylpyrazole, or (partially) etherified melamine-formaldehyde resins, as are described, for example, in Lackharze, Chemie, Eigenschaften und Anwendungen, Eds. D. Stoye, W. Freitag, Hanser Verlag, Munich, Vienna 1996.

Preference is given to the use of the aqueous or solvent-containing binders according to the invention in two-component systems together with polyisocyanates based on hexamethylene diisocyanate, isophorone diisocyanate, 4,4-diisocyanatodicyclohexylmethane, tetramethylene diisocyanate, 2-methylpentamethylene diisocyanate, 2,2,4- and 2,4,4-trimethylhexamethylene diisocyanate (THDI), 1,4-diisocyanato-cyclohexane, 3-isocyanatomethyl-1-methyl-1-isocyanatocyclohexane (IMCI), a,a,a',a'-tetramethyl-m- or -p-xylylene diisocyanate (TMXDI), 1,4- and 1,3-xylylene diisocyanate (XDI), hexahydroxylylene diisocyanate (H6-XDI) and/or mixtures thereof and optionally their variants rendered hydrophilic for aqueous binders, as are described, for example, in DE-A 41 36 618.

Particular preference is given, however, to polyisocyanates based on hexamethylene diisocyanate, isophorone diisocyanate and 4,4-diisocyanatodicyclohexylmethane.

Such polyisocyanates are used as hardeners in high-quality polyurethane lacquers which have outstanding resistance to chemicals and a high degree of gloss.

The conventional solvents are suitable for formulating the lacquers according to the invention, such as aromatic, aliphatic, araliphatic or cycloaliphatic hydrocarbons, partially or completely halogenated aromatic, aliphatic, araliphatic or cycloaliphatic hydrocarbons, alcohols, such as, for example, methanol, ethanol, isopropanol, butanol, benzyl alcohol, diacetone alcohol, esters, such as, for example, ethyl acetate, propyl acetate, butyl acetate, ether esters, such as, for example, methoxypropyl acetate or butyl glycol acetate, ketones, such as acetone, methyl ethyl ketone, methyl isobutyl ketone or cyclohexanone, strongly polar solvents, such as, for example, N,N-dimethylformamide, N,N-dimethylacetamide, dimethyl sulfoxite, N-methylpyrrolidone.

Further suitable solvents are water, liquid acid esters, such as, for example, phosphoric acid dibutyl ester, phosphoric acid tributyl ester, sulfonic acid esters, borates or derivatives of silica, for example tetraethoxysilane, methyltrimethoxysilane, 3-aminopropyl-trimethoxysilane, 3-aminopropyltriethoxysilane, glycidyl-oxypropyltrimethoxysilane, glycidyloxypropyl-triethoxysilane.

If those solvents are used as dispersing media for the silicas according to the invention, further substances may be added to those materials that are used. Preference is given to those substances which are also provided for subsequent further use or which improve the deagglomeration

or the properties, for example the stability, of the deagglomerated materials.

Particular preference is given to oligomeric or polymeric organic compounds, such as, for example, the above-mentioned resins and binders conventional in lacquers and coatings technology.

It is also possible to use in the dispersing medium amounts of the combination resins used in multi-component systems, such as, for example, the amine and melamine resins acting as hardeners, polyisocyanates, the adducts, referred to as blocked polyisocyanates, consisting of aromatic or aliphatic polyisocyanates and so-called cleaving agents, such as methyl ethyl ketoxime, caprolactam, malonic acid esters or triazole and 2,5-dimethylpyrazole. For procedures in which water is used as a substantial constituent of the dispersing medium, suitable additives are especially compounds which are soluble in or compatible with water, such as, for example, partially or completely saponified polyvinyl acetate, or variants of the above-mentioned classes of compounds which have been rendered hydrophilic. Further additives which are suitable and which are preferably used in aqueous media are, for example, silica sol and the sols of metallic oxides of the elements aluminium, titanium, zirconium, tantalum and tin.

As low molecular weight additives by means of which it is possible, for example, to achieve stabilisation of the deagglomerated silicas according to the invention against further agglomeration, there are suitable in principle all compounds which are used for stabilising nanoscale materials prepared by other means, for example by one of the methods mentioned above. Mention may be made of, for example, hydrolysable compounds carrying silane groups, such as alkoxysilanes or chlorosilanes. Particular amphiphilic compounds may also be used. The solvents and

additives that are suitable for the preparation of the dispersing medium can be mixed with one another as desired.

The lacquer binders according to the invention can be mixed with any conventional hardeners (combinations) to produce coatings.

The lacquer formulations according to the invention are transparent lacquer formulations containing pyrogenic, structurally modified silicas, the silicas according to the invention having an extremely small effect on the rheology of the lacquer formulation while at the same time exhibiting increased resistance of the lacquer surface to scratching.

The lacquer formulations according to the invention are particularly suitable for the production of clear lacquers, because significant improvements in the properties, such as an improvement in scratch resistance, can be achieved thereby while transparency and a high degree of gloss are retained. Typical clear lacquer applications in which the lacquer formulations according to the invention lead to particularly marked advantages are, for example, the series and repair lacquering of motor vehicles, and the wear-resistant coating of parquet floors and other floors.

In addition, owing to the structural modification, the pyrogenic silicas have a reduced tendency to dust formation, and incorporation into the lacquer binders is simplified.

The following two tables contain an overview of the production and the physicochemical data of the comparison silicas and the silicas according to the invention used in the Examples.

Table 1: Overview of the preparation of the comparison silicas and of the silicas according to the invention (Examples)

Description	Group attached to the surface	Structural modification	Milling after structural modification
Comparison silica 1	Hexadecylsilyl	No	-
Comparison silica 2	Octylsilyl	No	-
Comparison silica 3	Dimethylsilyl	No	-
Comparison silica 4	Dimethylsilyl	No	-
Silica 1	Hexadecylsilyl	Yes	Yes
Silica 2	Hexadecylsilyl	Yes	No
Silica 3	Octylsilyl	Yes	Yes
Silica 4	Octylsilyl	Yes	No
Silica 5	Dimethylsilyl	Yes	No
Silica 6	Dimethylsilyl	Yes	No
Silica 7	Dimethylsilyl	Yes	No
Silica 8	Dimethylsilyl	Yes	No

Table 2: Physicochemical data of the comparison silicas and of the silicas according to the invention (Examples)

Description	Tamped density [g/l]	Loss on drying [%]	Ignition loss [%]	pH value	C content [%]	DBP adsorption [%]	Specific BET surface area [m²/g]
Comparison silica 1	57	0.5	1.8	4.6	1.2	302	195
Comparison silica 2	51	0.6	6.8	5.3	5.4	236	175
Comparison silica 3	64	0.1	0.5	4.0	0.8	243	113
Comparison silica 4	67	0.5	0.6	4.8	1.0	256	165
Silica 1	123	0.7	2.6	6.0	1.4	208	197
Silica 2	146	1.1	2.3	5.8	1.4	192	195
Silica 3	240	0.8	6.7	4.8	5.3	87	169
Silica 4	322	0.3	6.9	6.0	5.3	not determinable	172
Silica 5	236	0.1	0.6	4.0	0.8	127	115
Silica 6	204	0.1	0.6	3.9	0.8	137	116
Silica 7	223	0.3	0.7	4.2	1.0	160	169
Silica 8	186	0.3	0.7	4.2	1.1	152	171

Example 1:

A conventional 2-component polyurethane lacquer was used for investigating the improvement in scratch resistance. The recipe for the lacquer and the preparation, including application, thereof are summarised hereinbelow:

Recipe 1:

Material to be ground	Parts by weight
Acrylate resin, 50 % in xylene/ethyl benzene 3:1	53.3
Butyl acetate 98 %	6.7
Xylene	6.7
Silica	5.0
Σ	71.7
Making up into a lacquer	
Acrylate resin, 50 % in xylene/ethyl benzene 3:1	1.1
Xylene	12.2
Ethoxypropyl acetate	1.5
Butyl glycol acetate	1.5
Butyl acetate 98 %	-
Aliphatic polyisocyanate, about 75 % in 1-methoxypropyl acetate-2/xylene 1:1	17.0
Σ	105.0

Binder concentration:	40 %
Silica calculated on material to be ground (FK):	18.8 %
Silica calculated on lacquer (total):	5.0 %
Silica calculated on lacquer (FK):	12.5 %

Preparation and application of the lacquers

The binder is mixed with the solvents. For preliminary dispersion, the silica is then incorporated into that mixture using a dissolver (plate Ø 45 mm) and pre-dispersed for 5 minutes at 2000 rpm. The mixture is dispersed in a laboratory bead mill for 30 minutes at 2500 rpm and 60 % pump capacity using glass beads (Ø about 1 mm). The dispersed material is checked using a grindometer, 25 µm, according to DIN ISO 1524. It must be smaller than 10 µm.

The ground material is made up into a lacquer according to the recipe, the components being mixed by means of a blade-type stirrer at 2000 rpm. The hardener is incorporated in the same manner.

After adjusting the lacquers to spraying viscosity according to DIN 53411, the lacquers are applied by means of spray application to black-lacquered metal sheets, e.g. DT 36 (Q-Panel) (layer thickness about 40-50 µm). After spraying, the metal sheets are dried for 24 hours at room temperature and then for 2 hours in a drying oven at 70°C.

Scratching tests:

By means of a scouring- and washing-resistance testing machine (Erichsen, pig-bristle brush), the metal sheets are scoured using a quartz/water suspension (100 g of water +

1 g of Marlon A 350, 0.25 % + 5 g of Sikron F500) and also using a $CaCO_3$/water mixture (100 g of water + 1 g of Marlon A 350, 0.25 % + 5 g of Millicarb BG). The gloss before scouring and 10 minutes after scouring is determined using a reflectometer (angle of irradiation 20°).

Table 3: next page

Because of their substantially lower rheological activity as compared with the comparison silicas, all the silicas according to the invention can be used in high concentrations without impairing the appearance of the lacquer surface. In addition, the silicas according to the invention exhibit a substantial improvement in the scratch resistance of the lacquer surfaces.

Table 3a: Summary of the relevant lacquer-related properties of the liquid lacquers and of the applied and dried films

	Comparison S1	S 1	S 2	Reference without S	Comparison S2	S 3	S 4	Reference without S
Grindometer value [µm]	< 10	< 10	< 10	< 10	< 10	< 10	< 10	/
Viscosity (ground material) [mPas]								
6 rpm	409	210	220	/	5670	935	832	/
60 rpm	407	210	212	/	1260	409	407	/
Viscosity (lacquer+hardener) [mPas]								
6 rpm	120	80	80	60	446	195	175	55
60 rpm	113	82	82	61	194	146	144	64
Flow	poor fine cracks	o. k.	o. k.	o. k.	orange-peel effect	o. k.	o. k.	o. k.
Scratch resistance								
20° reflectometer value before scratching	81	89.5	89.1	91.3	38	85.5	85.3	91.7
Haze before scratching	101	9	12	2	423	18	19	2
40 strokes with Sikron F 500 residual gloss [%]	83.4	88.5	90.7	51.8	/	80.4	84.3	56.1

Table 3b: Summary of the relevant lacquer-related properties of the liquid lacquers and of the applied and dried films

	Comparison S3	S 5	S 6	Reference without S	Comparison S4	S 7	S 8	Reference without S
Grindometer value [µm]	< 10	< 10	< 10	/	< 10	< 10	< 10	/
Viscosity (ground material) [mPas]								
6 rpm	4710	421	772	571	4990	802	772	55
60 rpm	1120	210	264	225	1200	279	264	52
Viscosity (lacquer+hardener) [mPas]								
6 rpm	882	105	210	135	857	235	105	70
60 rpm	239	75	92	79	242	100	26	37
Flow	orange-peel effect	o. k.	o. k.	o. k.	orange-peel effect	o. k.	o. k.	o. k.
Scratch resistance								
20° reflectometer value before scratching	39.0	83.4	83.5	88.8	67.2	84.2	82.6	89.3
Haze before scratching	430	30	40	3	235	18	10	2
40 strokes with Sikron F 500 residual gloss [%]	/	83.7	82.3	56.0	/	74.7	80.4	47.2

Example 2

In this Example, the effect of structural modification has been investigated with reference to a high solids 2K PUR clear lacquer. The recipe for the lacquer and the preparation, including application and testing, thereof are summarised hereinbelow:

Recipe 2:

Material to be ground	Parts by weight
Acrylic copolymer, modified with synthetic fatty acids, 70 % in n-butyl acetate	61.0
Butyl acetate 98 %	7.3
Methoxypropyl acetate	1.7
Solvesso 100	2.0
Xylene	2.0
Baysilon OL 17, 10 % in xylene (silicone oil)	0.7
Silica	5.0
Σ	79.7
Making up into a lacquer (hardener)	
Aliphatic polyisocyanate, 90 % in n-butyl acetate	22.3
Butyl acetate 98 %	2.0
Solvesso 100	1.0
Σ	105.0

Binder concentration:	62.8 %
Silica calculated on material to be ground (FK):	11.7 %
Silica calculated on lacquer (total):	5.0 %
Silica calculated on lacquer (FK):	8.0 %

Preparation and application of the lacquers

The binder is mixed with the solvents. For preliminary dispersion, the silica is then incorporated into that mixture using a dissolver (plate Ø 45 mm) and pre-

dispersed for 5 minutes at 2000 rpm. The mixture is dispersed in a laboratory bead mill for 30 minutes at 2500 rpm and 60 % pump capacity using glass beads (Ø about 1 mm). The dispersed material is checked using a grindometer, 25 µm, according to DIN ISO 1524. It must be smaller than 10 µm.

The ground material is made up into a lacquer according to the recipe, the components being mixed by means of a blade-type stirrer at 2000 rpm. The hardener is incorporated in the same manner.

After adjusting the lacquers to spraying viscosity according to DIN 53411, the lacquers are applied by means of spray application to black-lacquered metal sheets, e.g. DT 36 (Q-Panel) (layer thickness about 40-50 µm). After spraying, the metal sheets are dried for 24 hours at room temperature and then for 2 hours in a drying oven at 70°C.

Scratching tests:

By means of a scouring- and washing-resistance testing machine (Erichsen, pig-bristle brush), the metal sheets are scoured using a quartz/water suspension (100 g of water + 1 g of Marlon A 350, 0.25 % + 5 g of Sikron F500). The gloss before scouring and 10 minutes after scouring is determined using a reflectometer (angle of irradiation 20°).

Table 4: Summary of the relevant lacquer-related properties of the liquid lacquers and of the applied and dried films

	Comparison S1	S 1	S 2	Reference without S	Comparison S4	S 7	S 8	Reference without S
Grindometer value [µm]	< 10	< 10	< 10	/	< 10	< 10	< 10	/
Viscosity (ground material) [mPas]								
6 rpm	767	376	376	205	6200	1500	541	140
60 rpm	717	359	361	205	2100	900	559	195
Viscosity (lacquer+hardener) [mPas]								
6 rpm	459	279	281	120	3821	1041	497	167
60 rpm	399	272	274	120	1320	666	446	195
Flow	poor (fine "cracks")	o. k.	o. k.	o. k.	pronounced orange peel	o. k.	o. k.	o. k.
Scratch resistance								
20° reflectometer value before scratching	82.3	86.5	86.3	88.2	81.0	83.5	82.8	88.0
Haze before scratching	3	4	4	2	25	5	6	2
40 strokes with Sikron F 500 residual gloss [%]	63.2	78.2	75.4	30.2		94.3	93.4	82.0

Because of their substantially lower rheological activity as compared with the comparison silicas 1 and 4, the silicas 1 + 2 and 7 + 8 according to the invention can be used in high concentrations without impairing the appearance of the lacquer surface. In addition, the silicas according to the invention exhibit a substantial improvement in the scratch resistance of the lacquer surface.

Example 3:

A conventional 2-component polyurethane lacquer was used for investigating the improvement in scratch resistance. The recipe for the lacquer and the preparation, including application, thereof are summarised hereinbelow:

Recipe 3

Material to be ground	Parts by weight
Acrylic copolymer, modified with synthetic fatty acids, 60 % solution	43.4
Butyl acetate 98 %	17.8
Xylene	3.9
Silica	5.0
Σ	70.7
Making up into a lacquer	
Xylene	11.3
Ethoxypropyl acetate	3.4
Butyl glycol acetate	1.6
Aliphatic polyisocyanate, about 75 % in 1-methoxypropyl acetate-2/xylene 1:1	18.6
Σ	105.0

Binder concentration:	40 %
Silica calcuated on material to be ground (FK):	19.2 %
Silica calculated on lacquer (total):	5.0 %
Silica calculated on lacquer (FK):	12.5 %

Preparation and application of the lacquers

The binder is mixed with the solvents. For preliminary dispersion, the silica is then incorporated into that mixture using a dissolver (plate Ø 45 mm) and pre-dispersed for 5 minutes at 2000 rpm. The mixture is dispersed in a laboratory bead mill for 30 minutes at 2500 rpm and 60 % pump capacity using glass beads (Ø about 1 mm). The dispersed material is checked using a grindometer, 25 µm, according to DIN ISO 1524. It must be smaller than 10 µm.

The ground material is made up into a lacquer according to the recipe, the components being mixed by means of a blade-type stirrer at 2000 rpm. The hardener is stirred in in the same manner.

After adjusting the lacquers to spraying viscosity according to DIN 53411, the lacquers are applied by means of spray application to black-lacquered metal sheets, e.g. DT 36 (Q-Panel) (layer thickness about 40-50 µm). After spraying, the metal sheets are dried for 24 hours at room temperature and then for 2 hours in a drying oven at 70°C.

Scratching tests:

By means of a scouring- and washing-resistance testing machine (Erichsen, pig-bristle brush), the metal sheets are scoured using a quartz/water suspension (100 g of water + 1 g of Marlon A 350, 0.25 % + 5 g of Sikron F500). The gloss before scouring and 10 minutes after scouring is determined using a reflectometer (angle of irradiation 20°).

Table 5: see next page

Because of their substantially lower rheological activity as compared with the comparison silicas 1 and 2, the silicas 1 + 2 and 3 + 4 according to the invention can be used in high concentrations without impairing the appearance of the lacquer surface. In addition, the silicas according to the invention exhibit a substantial improvement in the scratch resistance of the lacquer surface.

Table 5: Summary of the relevant lacquer-related properties of the liquid lacquers and of the applied and dried films

	Comparison silica 1	Silica 1	Silica 2	Reference without silica	Comparison silica 2	Silica 3	Silica 4	Reference without silica
Grindometer value [µm]	< 10	< 10	< 10	/	< 10	< 10	< 10	/
Viscosity (ground material) [mPas]								
6 rpm	409	210	220	/	5670	935	832	/
60 rpm	407	210	212	/	1260	409	407	/
Viscosity (lacquer+hardener) [mPas]								
6 rpm	120	80	80	60	446	195	175	55
60 rpm	113	82	82	61	194	146	144	64
Flow	poor fine cracks	o. k.	o. k.	o. k.	orange-peel effect	o. k.	o. k.	o. k.
Scratch resistance								
20° reflectometer value before scratching	81	89.5	89.1	91.3	38	85.5	85.3	91.7
Haze before scratching	101	9	12	2	423	18	19	2
40 strokes with Sikron F 500 residual gloss	83.4	88.5	90.7	51.8	/	80.4	84.3	56.1

Example 4

Direct comparison of the silicas according to the invention with a scratch-resistant lacquer according to DE 198 11 790 A1, in which AEROSIL R 972 is used to improve the scratch resistance.

Recipe 4

	Prior art 1)	**Silicas according to the invention 2)**
Material to be ground		
Desmophen A 2009/1		190.2
Methoxypropyl acetate : Solvesso 100 1:1		36.8
Silica		23.0
Σ		250.0
Making up into a lacquer		
Desmophen A YEP4-55A, contains AEROSIL R 972	96.0	-
Ground material	-	48.9
Desmophen 2009/1	-	24.9
OL 17, 10 % in MPA	-	-
Modaflow 1 % in MPA	-	-
MPA : Solvesso 100 1:1	11.6	33.8
Butyl glycol acetate	10.5	10.5
Byketol OK	7.5	7.5
Byk 141	0.8	0.8
Addition of hardener		
Desmodur N 3390	23.6	23.6
Σ	150.0	150.0

Preparation and application of the lacquers

1) The comparison silica 1 is incorporated into the binder according to DE 198 11 790 A1 by means of a jet disperser.

2) The binder is mixed with the solvents. For preliminary dispersion, the silica is then incorporated into that mixture using a dissolver (plate Ø 45 mm) and pre-dispersed for 5 minutes at 2000 rpm. The mixture is dispersed in a laboratory bead mill for 30 minutes at 2500 rpm and 60 % pump capacity using glass beads (Ø about 1 mm). The dispersed material is checked using a grindometer, 25 µm, according to DIN ISO 1524. It must be smaller than 10 µm.

The ground material is made up into a lacquer corresponding to 1) or 2) according to the recipe, the components being mixed by means of a blade-type stirrer at 2000 rpm. The hardener is stirred in in the same manner.

After adjusting the lacquers to spraying viscosity according to DIN 53411, the lacquers are applied by means of spray application to black-lacquered metal sheets, e.g. DT 36 (Q-Panel) (layer thickness about 40-50 µm). After spraying, the metal sheets are dried for 24 hours at room temperature and then for 2 hours in a drying oven at 70°C.

Scratching tests:

By means of a scouring- and washing-resistance testing machine (Erichsen, pig-bristle brush), the metal sheets are scoured using a $CaCO_3$/water suspension (100 g of water + 1 g of Marlon A 350, 0.25 % + 5 g of $CaCO_3$ Millicarb). The gloss before scouring and 10 minutes after scouring is determined using a reflectometer (angle of irradiation 20°).

Table 6: Summary of the relevant lacquer-related properties of the liquid lacquers and of the applied and dried films

	Prior art	Silica 1	Silica 5	Silica 8	Reference without S
Grindometer value [µm]	< 10	< 10	< 10	< 10	/
Viscosity (ground material)					
6 rpm	58	30	30	26	30
60 rpm	48	43	43	33	40
Surface	orange peel	o.k.	o.k.	o.k.	o.k.
20° reflectometer value before scratching	88.0	86.5	90.2	89.6	98.5
100 strokes with Millicarb residual gloss	88.3	96.3	95.4	97.1	59.6

It is found that, by the use of the silicas according to the invention, a substantially greater improvement in the residual gloss after scratching of the lacquer surface is achieved than in the case of the prior art. In addition, owing to their low rheological activity, the silicas according to the invention do not cause an orange-peel effect.